# *Analytical Considerations for the Determination of Beryllium on Air Filters and Surface Smears Using High Throughput Automated Fluorescence*

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- Instrumentation and analysis costs

# *Fluorescence method background*

Approved methods:

- ASTM D7202
- NIOSH 7704
- NIOSH 9110

Basic methods designed for field use, or small sample load.

Methods utilize manual, sequential sample analysis approach.

# *Other analysis methods*

- ICP-OES
- ICP-MS
- Graphite furnace AAS

These techniques also employ a sequential sample analysis approach:

Sample#1…Sample#2…Sample#3…

# Automated Fluorescence

- Utilizes robotic liquid handling system and microplate reader instrument.
- Analysis of 96 well plate possible in < 3 minutes.
- Excellent detection limits (MDL=0.00079 µg/wipe)
- Large analytical range (0.01µg - 20 µg)
- Highly specific for beryllium, few analytical interferences

# QC – an interesting situation

- The well plate reader instrument approach created a need for an alternative approach to run level quality control.

- Existing lab instrumentation and methods are sequentially based, i.e. one sample at a time covering long periods of time (3-5 hours).

- Instrument drift is a primary concern and frequent calibration verification standards are employed by most methods to verify the standardization on an on-going basis.

# QC – automated fluorescence approach

- Full tray of 96 samples read in less than 3 minutes.
- Calibration drift is not a concern.
- Design a QC protocol to verify fitness of use, while maximizing instrument efficiency and productivity.
- Follow guidance from AIHA and A2LA lab accreditation checklists.

# QC protocol for automated fluorescence

- Initial calibration verification (ICV) using a second source standard
- Blank level checking: calibration (CCB) and method blanks
- Reporting limit verification
- Duplicate lab controls (aqueous or BeO)
- Method precision from LCS duplicates
- End-of-plate calibration verification (CCV)

# 96 well plate layout - QC

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A | Blank | Blank | ICV | | | | | | | | | |
| B | Std #1 | Std #5 | ICB | | | | | | | | | |
| C | Std #2 | Std #6 | RLV | | | | | | | | | |
| D | Std #3 | Std #7 | Well left empty | | | | | | | | | |
| E | Blank | Blank | Method blank#1 | | | | | | | | | Method blank#2 |
| F | Std #4 | Std #8 | LCS-1 | | | | | | | | | LCS-2 |
| G | Std #5 | Std #9 | | | | | | | | | | CCB |
| H | Blank | Blank | | | | | | | | | | CCV |

# Y-12 ICP method

Batch size = 45 air/smears

Sample digestion time = 63 minutes
ICP determination time = 180 minutes
Total analysis time = 243 minutes

Per sample time = 5.4 minutes/sample

# Automated fluorescence method

Batch size = 70 air/smears

Sample prep time = 140 minutes
Fluorescence determination time = 2 minutes
Total analysis time = 147 minutes

Per sample time = 2.1 minutes/sample

# Advantages

- Robotic liquid handling system reduces ergonomic issues with manual prep
- Instrumentation requires little maintenance
- Allows very large batches for increased capacity
- 4-tip system: 400 samples/8 hr shift
- Decreased turnaround time by about 50%
- Overall analytical costs decrease from ICP method

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